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(21) International Application Number: PCT/GB99/04424 (22) International Filing Date: 23 December 1999 (23.12.99) (30) Priority Data: 9828445.8 24 December 1998 (24.12.98) GB (71) Applicant (for all designated States except US): PPG INDUSTRIES OHIO, INC. [US/US]; 3800 West 143rd Street, Cleveland, OH 44111 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): FENN, David, Robert [GB/GB]; 29 Wendover Road, Burnham, Buckinghamshire SL1 7ND (GB). LAM, Peter, Kwok, Hing [GB/DK]; Klørvang 35, DK-2970 Hørsholm (DK). (74) Agent: W.P. THOMPSON & CO.; Coopers Building, Church Street, Liverpool L1 3AB (GB).		(81) Designated States: AU, BR, CA, JP, MX, US, ZA, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: COATING COMPOSITION (57) Abstract A coating composition comprising: i) diphenylmethane diisocyanate, and ii) a hydroxyl functional compound which is a polyester having secondary hydroxyl groups, the secondary hydroxyl groups being the product of a reaction between a carboxylic acid group and an epoxide group, the composition containing substantially no reactive diluent which are aldimines, ketimines or aspartic esters.		

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Coating Composition

This invention relates to polyurethane coating compositions, their preparation and use.

Coatings used for the repair and refinishing of vehicles such as motorcars (known
5 as vehicle refinish paints) are required to have very good physical properties such as hardness and resistance to water and solvents. The coating compositions are also required to have good application and appearance properties so that they are easy to apply to a substrate using spray application and result in final films having a good gloss.

One class of coating compositions commonly used for these applications
10 comprises a hydroxyl functional polymer, such as a polyester or acrylic polymer, and a polyisocyanate. These two components react together after application to a substrate to form a polyurethane coating. These compositions are often thus referred to as 'polyurethane coating compositions' although strictly the polyurethane is only formed when the coating crosslinks.

15 The two components of polyurethane coating compositions begin to react together as soon as they are mixed and for this reason these compositions are usually supplied in the form of two components which are mixed together shortly before use. Because of the

reaction between the components, the viscosity of the mixture begins to rise slowly right from first mixing. The mixed compositions have a limited useful life, referred to as the 'pot-life' after which the viscosity rise makes them unusable. Eventually, the mixed compositions in bulk become solid, or semi-solid. Typical pot-lives for vehicle refinish polyurethane coating compositions range from 30 minutes to 5 hours, typically 30 minutes to 3 hours.

Several different types of polyisocyanate are known for use in these types of composition, including the isocyanurate trimers of hexamethylene diisocyanate, and isophorone diisocyanate and polyisocyanate prepolymers made by reaction of these diisocyanates and others such as toluene diisocyanates with polyols such as trimethylol propane.

One other type of polyisocyanate is diphenylmethane diisocyanate, a mixture of 2 isomers, 4,4- and 2,4-, also known as MDI. MDI is widely used in producing polyurethane foams. MDI is available in several forms, based on two types of product, purified monomeric MDI and polymeric MDI. MDI is relatively cheap amongst polyisocyanates and polymeric MDI is a low viscosity liquid at room temperature. In these respects MDI would be ideal for use in polyurethane coating compositions for vehicle refinish paints, particularly for primers. However, the isocyanate groups on MDI are very reactive and it has been found impossible to simply use MDI as a direct replacement for the other useful polyisocyanates in these types of composition because the resulting compositions have too short a pot-life. Thus, it has not been realistically possible to use MDI in practical refinish paints even though it has several attractive properties.

We have now discovered that it is possible to formulate practical polyurethane coating compositions which contain MDI by using a particular hydroxyl containing component having secondary hydroxyl groups, the secondary hydroxyl groups being the product of a reaction between a carboxylic group and an epoxide group. This type of hydroxyl containing component has itself been suggested for use in polyurethane vehicle refinish paints but only in conjunction with a reactive diluent having blocked primary amine groups or hindered secondary amine groups which are ketimines, aldimines or aspartic esters. These reactive diluents are expensive and this is a disincentive to the use of these compositions. Furthermore these previously proposed compositions often have to be formulated as

three-pack compositions, the packs containing polymer, polyisocyanate and reactive diluent respectively, which makes them more complicated for the end-user to make up into the final paint. In contrast, the compositions of the present invention both avoid the use of expensive reactive diluents and utilise relatively low cost polyisocyanate to make two-pack polyurethane compositions. These coating compositions have a good pot-life and which also give films having good properties. The compositions are particularly useful as primers for vehicle refinish.

According to the present invention there is provided a coating composition comprising ;

- i) diphenylmethane diisocyanate, and
 - ii) a hydroxyl functional compound which is a polyester having secondary hydroxyl groups, the secondary hydroxyl groups being the product of a reaction between a carboxylic acid group and an epoxide group,
- the composition containing substantially no reactive diluent which are aldimines, ketimines or aspartic esters.

A further benefit of using diphenylmethane diisocyanate (MDI) is that the compositions do not require the presence of a reactive diluent to lower the viscosity of the composition, or to increase the speed of reaction of the components.

MDI is commercially available, for example as Suprasec MDI and MDI-based prepolymers from ICI Polyurethanes. MDI is preferably used in an amount such that the ratio of isocyanate groups on the MDI to the total number of hydroxyl groups on the hydroxyl functional compound is in the range 0.7:1 to 3:1, more preferably 1:1 to 3:1.

Preferably the hydroxyl functional compound has a molecular weight of less than 5000, more preferably less than 2500 and most preferably less than 2000.

Preferably the hydroxyl functional compound is the reaction product of a polyfunctional carboxylic acid and a monoepoxide, and so has secondary hydroxyl groups

arising from the reaction of the acid groups on the polycarboxylic acid with the epoxy group on the monoepoxide.

Preferred monoepoxides are glycidyl esters of C2-C20 alkanolic acids, preferably C8-C9 tertiary carboxylic acids such as versatic acid. One particularly suitable
5 monoepoxide is Cardura E10TM.

The polyfunctional carboxylic acid can be a polyester with two or more carboxylic acid groups, preferably two to four carboxylic acid groups, more preferably two or three such groups. Polyesters are compounds containing more than one ester group. Acid functional polyesters can be made by conventional methods from a mixture of polyols,
10 diacids and anhydrides. One preferred acid functional polyester can be made by reaction of a polyol with a carboxylic acid anhydride. Polyols are compounds with more than one hydroxyl group. Preferably the polyol has from two to six hydroxyl groups per molecule. Preferably the polyol has a molecular weight of 66 to 150. Examples of suitable polyols are ethylene glycol, propylene glycol, 1,4-butane diol, neopentyl glycol, hexane-1,6-diol,
15 glycerol, trimethylol propane and pentaerythritol. Preferred anhydrides are cyclic internal anhydrides of dicarboxylic acids such as maleic anhydride, succinic anhydride, phthalic anhydride and hexahydroxyphthalic anhydride.

Preferably the reaction between the anhydride and the polyol is carried out substantially stoichiometrically so that one anhydride molecule reacts with each hydroxyl
20 group on the polyol. The resulting polyfunctional acid compound has substantially the same number of acid groups as the polyol had hydroxyl groups. Because the hydroxyl/anhydride reaction occurs at a lower temperature than the carboxyl/hydroxyl or transesterification reactions, there are few side reactions and the molecular weight of the product can be controlled.

25 The reaction between the polyol and the anhydride can be carried out by dissolving the components in an organic solvent and then heating the solution preferably to between about 100 and 130°C for between about 1 and 3 hours so as to produce the polyfunctional carboxylic acid.

The reaction between the polyfunctional carboxylic acid and the monoepoxide can be carried out by adding the monoepoxide to a solution of the polycarboxylic acid in a suitable organic solvent in an amount so as to react each of the hydroxyl groups on the polyol with an epoxide group, and heating the mixture to between about 130 and 160°C for between about 2 and 12 hours.

The compositions can optionally contain a volatile organic solvent. The volatile organic solvent can be any solvent which will dissolve the polyisocyanate, and the hydroxyl functional compound. It can be an aliphatic or aromatic hydrocarbon such as Solvesso 100™, toluene or xylene, an alcohol such as butanol or isopropanol, an ester such as butyl acetate or ethyl acetate, a ketone such as acetone, methyl isobutyl ketone or methyl ethyl ketone, an ether, an ether-alcohol or an ether-ester or a mixture of any of these.

Preferably the compositions contain less than 500 g/l of volatile organic solvent based on the total composition, more preferably less than 480 g/l still more preferably less than 420g/l and most preferably less than 250g/l.

The compositions can also contain catalysts for the isocyanate-hydroxyl reaction. Suitable catalysts include tin catalysts such as dibutyl tin dilaurate and amine catalysts such as triethylamine. The compositions can also contain other conventional paint additives such as, pigments, fillers, UV absorbers and flow aids. Preferably the composition is pigmented, that is, it also contains pigments and optionally fillers. Most preferably, the composition is pigmented so as to be useful as a vehicle refinish primer.

The compositions can be made by mixing the components, optionally dissolved in organic solvent, in any order.

The coating composition of the invention can be applied to the surface of a substrate and then allowed or caused to dry and cure. According to the present invention there is provided a process for coating a substrate which comprises the steps of applying a layer of a coating composition according to the present invention to a surface of the substrate and thereafter causing or allowing the layer to cure.

The coating composition can be applied by conventional means such as by brush, roller or spray, preferably spray. The substrate can be for example, metal, plastic, wood or glass. The compositions are particularly useful for refinishing motor vehicles, and especially as primers.

5 The applied layer can be allowed to cure at ambient temperature in the case where the hydroxyl polymer and crosslinker react together at ambient temperatures. Alternatively the layer can be baked at elevated temperatures, for example 50-120°C to accelerate curing. Drying and curing typically takes between 5 minutes and 24 hours depending on the ambient conditions and on the particular components used. Conveniently it takes about 10 15 minutes to about 5 hours.

According to the present invention there is also provided a coated article obtainable by the process.

The invention will now be illustrated by means of the following examples.
The MDI used in the samples was Suprasec DNR from ICI Polyurethanes.

EXAMPLES

15 Polyester 1

A mixture of neopentyl glycol (5.1 parts), 1,4-butanediol (4.5 parts) and hexahydroxy phthalic anhydride (30.4 parts) were heated under nitrogen to about 95°C so as to melt the components. The molten mixture was stirred and the temperature was slowly raised until an exotherm occurred at about 130°C. At this point, heating was stopped and the 20 temperature went up to 170°C, then down to 150°C whereupon heating was resumed and the mixture was held at 150°C for 1 hour. The mixture was then allowed to cool for 1 hour. The mixture was heated back up to 150°C, Cardura E10™ (glycidyl ester of a C8-9 carboxylic acid from Shell, 50.0 parts) was added dropwise over a period of 3.5 hours and the mixture was allowed to cool to room temperature and left overnight. The mixture was 25 heated to 150°C for a further 7 hours before being allowed to cool to room temperature. Butyl acetate (10 parts) was added to give a theoretical non-volatile solids content of 90%. The final measured acid value was 0.5 mgKOH/g based on non-volatile content. The number average molecular weight as measured by G.P.C. was 914.

Primer composition

The following were added slowly in the order given to a mixture of Polyester 1 from above (27.8 parts), methyl isoamyl ketone (MIAK, 18.5 parts) and Byk 220S dispersant (2.78 parts), bentone (0.91 parts), talc (43 parts), extender (34.4 parts), titanium dioxide (43.3 parts), zinc phosphate pigment ZPA (18.2 parts), black tinter (2.3 parts) and defoamer (2.8 parts). The mixture was dispersed for a further 20 minutes. Further MIAK (6.0 parts) was added to form a pigmented primer base.

Polyester 1 (41.4 parts) and MDI (33.9 parts) were added to the primer composition and the mixture was thinned to 25 seconds BSB4 with MIAK.

- 10 The viscosity of the bulk composition was measured using a BSB4 cup 0.5 hours after making it as a guide to its pot-life to be 29 seconds.

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Polyester 2

A mixture of neopentyl glycol (5.6 parts), trimethylol propane (4.8 parts) and hexahydroxy phthalic anhydride (33.2 parts) were heated under nitrogen to about 95°C so as to melt the components. The molten mixture was stirred and the temperature was slowly raised until an exotherm occurred at about 130°C. At this point, heating was stopped and the temperature went up to 165°C, then down to 150°C whereupon heating was resumed and the mixture was held at 150°C for 1 hour. Cardura E10TM (glycidyl ester of a C8-9 carboxylic acid from Shell, 48.6 parts) was added dropwise over a period of 3.5 hours and the mixture was allowed to cool to room temperature and left overnight. The mixture was heated to 150°C for a further 7 hours before being allowed to cool to room temperature. Butyl acetate (7.7 parts) was added. The final measured acid value was 16.8 mgKOH/g based on non-volatile content. The number average molecular weight as measured by G.P.C. was 870.

Polyester 3

Stage 1

Adipic acid (49.7 parts), trimethylol propane (13.0 parts) and neopentyl glycol (30.4 parts) were heated with stirring under a nitrogen atmosphere until the mixture began to exotherm (at about 95°C) when heating was stopped. The temperature of the mixture rose to about 135°C due to the exotherm. When the exotherm was over and the temperature began to fall, heating was resumed and the temperature was raised to 205°C. As the temperature was raised, water was removed via a fractionating column. When the mixture was at 205°C and the evolution of water had slowed, the mixture was allowed to cool.

Stage 2

The fractionating column was replaced with a Dean & Stark column filled with methyl isobutyl ketone. Methyl isobutyl ketone (1.4 parts) was added to the mixture and the temperature was raised to reflux to remove further water. When the acid value of the mixture reached 15 mgKOH/g non-vol. the heat was removed. The temperature was allowed to fall to 100 °C and then butyl acetate (5.5 parts) was added. The number average molecular weight as measured by G.P.C. was 1900.

Primer composition 2

The following were added slowly in the order given to a mixture of Polyester 2 from above (13.27 parts), methyl isoamyl ketone (MIAK, 9.27 parts) and Byk 220S dispersant (1.40 parts). Bentone (0.45 parts), silica extender (0.74 parts), calcium magnesium carbonate (18.75 parts), magnesium silicate (11.48 parts), zinc phosphate pigment ZPA (12.00 parts), titanium dioxide (27.51 parts) black tinter (1.24 parts) and defoamer (1.41 parts). The mixture was dispersed for a further 20 minutes. Further MIAK (1.79 parts) and Polyester 2 (23.196 parts) were added to form a pigmented primer base.

MDI (Suprasec DNR available from ICI Polyurethanes, 10.26 parts) was added to the primer composition and the mixture was thinned to 25 seconds BSB4 with butyl acetate.

The viscosity of the bulk composition was measured using a BSB4 cup 1 hour after making it as a guide to its pot-life.

Primer composition 3

The following were added slowly in the order given to a mixture of Polyester 3 from above (13.27 parts), methyl isoamyl ketone (MIAK, 9.27 parts) and Byk 220S dispersant (1.40 parts). Bentone (0.45 parts), silica extender (0.74 parts), calcium magnesium carbonate (18.75 parts), magnesium silicate (11.48 parts), zinc phosphate pigment ZPA (12.00 parts), titanium dioxide (27.51 parts) black tinter (1.24 parts) and defoamer (1.41 parts). The mixture was dispersed for a further 20 minutes. Further MIAK (1.79 parts) and Polyester 3 (23.196 parts) were added to form a pigmented primer base.

MDI (Suprasec DNR available from ICI Polyurethanes, 10.26 parts) was added to the primer composition and the mixture was thinned to 25 seconds BSB4 with butyl acetate.

The viscosity of the bulk composition was measured using a BSB4 cup 1 hour after making it as a guide to its pot-life.

Primer compositions 2 and 3 were tested by spray applying the primer to cold rolled steel test panels, testing the primers for dust free time, print free time and hardness, and topcoating with a colourcoat and then evaluating the test panels for cross hatch adhesion and appearance. Four test panels were sanded with P180 sandpaper and solvent wiped

with white spirit. The panels were coated with 20-25 microns of chromate free etch primer P565-713 (available from ICI Autocolor, used as instructed on the product data sheet). Primer 2 and primer 3 were each spray applied on to two of the panels to give a film build of 100-120 microns. One panel coated with each primer was used for the evaluations shown in the table below.

	Initial viscosity (/secs BSB4 cup)	Viscosity after 1 hour (/secs BSB4 cup)	Solids at 25 seconds BSB4 cup viscosity	Dust free time (/minutes)	Print free time (/minutes)	Konig hardness	
						1 hour	1 day
Primer 2	25	38	65.3%	40	90	32	64
Primer 3	26	66	58.3%	35	90	13	19

The dust free time (DFT) of each system was determined by dropping dust on to the panel at five-minute intervals. The DFT was the time after application when the dust could be removed using a small brush without marking the coating. The print free time (PFT) was determined by placing a 1" disc of Whatmann No. 1 filter paper on the drying panel under a 1" rubber bung and a 50 g weight. The filter paper is held in place for exactly sixty seconds and the weight and bung are then removed. The panel is then lifted and inverted. The PFT was the first time when the filter paper falls off without leaving a mark on the coating.

The results show that primer 2 has a longer pot-life and better hardness development than primer 3.

The remaining panels were left at room temperature for four hours and then sanded with P360 sandpaper. The panel coated with primer 2 flatted easily. The panel coated with primer 3 was soft and difficult to flat. 100 parts by weight of Aquabase medium coarse aluminium basic P968-9987 was mixed with 10.3 parts by weight of Aquabase activator P935-2018 and 3 parts by weight of Aquabase thinner P275-366 (all available from ICI Autocolor). This was applied to both panels to give a film build of 12-15 microns. Both panels were then coated with P190-670 clearcoat (available from ICI Autocolor, used as instructed on the product data sheet). Both panels had excellent appearance and there was no discernible deterioration in appearance over one month. Crosshatch adhesion was

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measured according to ASTM D3359 one day and one month after application. Both panels gave 100% adhesion.

CLAIMS

1. A coating composition comprising;
 - i) diphenylmethane diisocyanate, and
 - ii) a hydroxyl functional compound which is a polyester having secondary hydroxyl groups, the secondary hydroxyl groups being the product of a reaction between a carboxylic acid group and an epoxide group, the composition containing substantially no reactive diluent which are aldimines, ketimines or aspartic esters.
2. A composition as claimed in Claim 1 in which the ratio of the isocyanate groups on the diphenylmethane diisocyanate to the total number of hydroxyl groups on the hydroxyl functional compound is 0.7:1 to 3.1.
3. A composition as claimed in claim 1 or claim 2 in which the hydroxyl functional compound has a molecular weight of less than 5000.
4. A composition as claimed in any one of claims 1 to 3 in which the hydroxyl functional compound is the reaction product of a polyfunctional carboxylic acid and a monoepoxide.
5. A composition as claimed in claim 4 in which the monoepoxide is a glycidyl ester of a C8-C9 tertiary carboxylic acid.
6. A composition as claimed in claim 4 or claim 5 in which the polyfunctional carboxylic acid is a polyester with two or more carboxylic acid groups.
7. A composition as claimed in any one of claims 4 to 6 in which the polyfunctional carboxylic acid is the reaction product of a polyol and an anhydride.

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8. A composition as claimed in Claim 7 in which the polyol has from 2 to 4 hydroxyl groups.
9. A composition as claimed in any one of claims 1 to 8 which is pigmented.
10. A process for preparing a composition as claimed in Claim 1 which comprises mixing
 - i) diphenylmethane diisocyanate, and
 - ii) a hydroxyl functional compound which is a polyester having secondary hydroxyl groups, the secondary hydroxyl groups being the product of a reaction between a carboxylic acid group and an epoxide group, optionally dissolved in organic solvent.
11. A process for coating a substrate which comprises the steps of applying a layer of a coating composition as claimed in any one of claims 1 to 9, to a surface of the substrate and thereafter causing or allowing the layer to cure.
12. A coated substrate obtainable by the process of Claim 11.

INTERNATIONAL SEARCH REPORT

Int. Application No.

PCT/GB 99/04424

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08G18/42 C08G18/76 C09D175/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08G C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 24 39 276 A (BASF AG) 26 February 1976 (1976-02-26) page 3, paragraph 2 -page 5, paragraph 1 claim 1	1,3,10
A	WO 97 30099 A (ICI PLC ;FENN DAVID ROBERT (GB)) 21 August 1997 (1997-08-21) page 3, line 1 -page 8, line 14 page 12, line 10 -page 13, line 26; example 1; table 1	1,5-8, 10-12
	— — — — — -/-	

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

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Date of the actual completion of the international search

1 March 2000

Date of mailing of the international search report

23/03/2000

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INTERNATIONAL SEARCH REPORT

Int. Application No.

PCT/GB 99/04424

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	NL 9 201 868 A (DSM NV) 16 May 1994 (1994-05-16) page 1, line 27 -page 5, line 13 claims 1,2 & DATABASE WPI Derwent Publications Ltd., London, GB; AN 94-189602 abstract	1,3-6, 10-12
A	WO 96 20968 A (DU PONT) 11 July 1996 (1996-07-11) page 4, line 14 -page 12, line 19 examples 1,35	1,3-8, 10-12

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 99/04424

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
DE 2439276	A	26-02-1976	NONE	
WO 9730099	A	21-08-1997	AU 1722597 A EP 0880550 A US 5977246 A	02-09-1997 02-12-1998 02-11-1999
NL 9201868	A	16-05-1994	NONE	
WO 9620968	A	11-07-1996	AU 4653196 A CA 2209230 A DE 69603153 D DE 69603153 T EP 0801661 A US 5753756 A	24-07-1996 11-07-1996 12-08-1999 18-11-1999 22-10-1997 19-05-1998

PATENT COOPERATION TREATY

From the:
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PCT

WRITTEN OPINION

(PCT Rule 66)

Date of mailing
(day/month/year) 06.09.2000

Applicant's or agent's file reference

JDM/P. 401106 WO

REPLY DUE

within 3 month(s)
from the above date of mailing

International application No.

PCT/GB99/04424

International filing date (day/month/year)

23/12/1999

Priority date (day/month/year)

24/12/1998

International Patent Classification (IPC) or both national classification and IPC

C08G18/42

Applicant

PPG INDUSTRIES OHIO, INC. et al.

RESPONSE DUE 6.12.00

1. This written opinion is the first drawn up by this International Preliminary Examining Authority.
2. This opinion contains indications relating to the following items:
 - I ☒ Basis of the opinion
 - II ☐ Priority
 - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
 - IV ☐ Lack of unity of invention
 - V ☒ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
 - VI ☐ Certain document cited
 - VII ☐ Certain defects in the international application
 - VIII ☐ Certain observations on the international application
3. The applicant is hereby **invited to reply** to this opinion.

When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also: For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.
4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 24/04/2001.

Name and mailing address of the international preliminary examining authority:



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WRITTEN OPINION

International application No. PCT/GB99/04424

I. Basis of the opinion

1. This opinion has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".*):

Description, pages:

1-11 as originally filed

Claims, No.:

1-12 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-4,10,11 (no)
Inventive step (IS)	Claims	5-9,12 (no)
Industrial applicability (IA)	Claims	1-12 (yes)

2. Citations and explanations

see separate sheet

Re Item V

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Novelty: DE-A-2 439 276 (D1) discloses reaction products of diphenyl methane-diisocyanate (MDI) and epoxide modified polyester polyols. The latter are reaction products of conventional polyesterpolyols and monoepoxides. The resulting epoxide modified polyester polyol, although not specifically mentioned in D1, necessarily carries secondary hydroxyl groups which result from the epoxide after ring opening. It should be noticed that according to the example in D1, the polyester polyol has an acid number, from which it is to be concluded that it has a certain number of pendant carboxylic acid groups, which are reactive with the monoepoxide yielding reaction products which correspond to those claimed in claims 1-4, 10 and 11 of the present application (Article 33,2 PCT).

Inventive step: Object of the present application was to provide MDI coating compositions showing the right reactivity and viscosity pattern and wherein the viscosity adjustment should be achieved without the use of aldimine, ketimine or aspartic ester reactive diluents. For a new claim to be filed it needs to be demonstrated that the distinguishing feature over D1 solves the object in a non-obvious way. From D1 it was apparent that epoxide modified polyesters are less reactive towards MDI, such that their evaluation in the design of coating compositions of controlled reactivity appears to be obvious (Article 33,3 PCT).

Industrial applicability: no objection.



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☎ +49 89 2399-0
TX 523 656 epmu d
FAX +49 89 2399-4465

Europäisches
Patentamt

Generaldirektion 2

European
Patent Office

Directorate General 2

Office européen
des brevets

Direction Générale 2

Correspondence with the EPO on PCT Chapter II demands

In order to ensure that your PCT Chapter II demand is dealt with as promptly as possible you are requested to use the enclosed self-adhesive labels with any correspondence relating to the demand sent to the Munich Office.

One of these labels should be affixed to a prominent place in the upper part of the letter or form etc. which you are filing.

INTERNATIONAL COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference JDM/P. 401106 WO	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/GB 99/ 04424	International filing date (day/month/year) 23/12/1999	(Earliest) Priority Date (day/month/year) 24/12/1998
Applicant PPG INDUSTRIES OHIO, INC.et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☐ Unity of invention is lacking (see Box II).

4. With regard to the title,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☒ None of the figures.

PATENT COOPERATION TREATY

PCT

NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

From the INTERNATIONAL BUREAU

To:

W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
ROYAUME-UNI

14 JUL 2000

LIVERPOOL

Date of mailing (day/month/year) 06 July 2000 (06.07.00)		
Applicant's or agent's file reference JDM/P. 401106 WO		
IMPORTANT NOTICE		
International application No. PCT/GB99/04424	International filing date (day/month/year) 23 December 1999 (23.12.99)	Priority date (day/month/year) 24 December 1998 (24.12.98)
Applicant PPG INDUSTRIES OHIO, INC. et al		

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:
AU,JP,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:
BR,CA,EP,MX,ZA

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on
06 July 2000 (06.07.00) under No. WO 00/39180

REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

<p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No. (41-22) 740.14.35</p>	<p>Authorized officer</p> <p>J. Zahra</p> <p>Telephone No. (41-22) 338.83.38</p>
---	--

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

**NOTIFICATION OF RECEIPT
OF SEARCH COPY**

(PCT Rule 25.1)

To:

W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
UNITED KINGDOM

Date of mailing
(day/month/year)

31/01/2000

Applicant's or agent's file reference

JDM/P. 401106 WO

IMPORTANT NOTIFICATION

International application No.

PCT/GB 99/04424

International filing date(day/month/year)

23/12/1999

Priority date (day/month/year)

24/12/1998

Applicant

PPG INDUSTRIES OHIO, INC.et al.

1. Where the International Searching Authority and the Receiving Office are not the same office:

The applicant is hereby notified that the search copy of the international application was received by this International Searching Authority on the date indicated below.

Where the International Searching Authority and the Receiving Office are the same office:

The applicant is hereby notified that the search copy of the international application was received on the date indicated below.

19/01/2000

(date of receipt).

2. ☐ The search copy was accompanied by a nucleotide and/or amino acid sequence listing in computer readable form.

3. Time limit for establishment of International Search Report

The applicant is informed that the time limit for establishing the International Search Report is 3 months from the date of receipt indicated above or 9 months from the priority date, whichever time limit expires later

4. A copy of this notification has been sent to the International Bureau and, where the first sentence of paragraph 1 applies, to the Receiving Office.

Name and mailing address of the International Searching Authority



European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl
Fax: (+31-70) 340-3016

Authorized officer

ISA/EP

PCT

From the INTERNATIONAL BUREAU

**NOTIFICATION OF THE RECORDING
OF A CHANGE**

(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)

To:

W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
ROYAUME-UNI

Date of mailing (day/month/year) 14 April 2000 (14.04.00)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference JDM/P. 401106 WO	
International application No. PCT/GB99/04424	International filing date (day/month/year) 23 December 1999 (23.12.99)

1. The following indications appeared on record concerning:

☒ the applicant ☒ the inventor ☐ the agent ☐ the common representative

Name and Address LAM, Peter, Kwok, Hing 22 Glenwood Road Hounslow Middlesex TW3 1SW United Kingdom	State of Nationality	State of Residence GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

☐ the person ☐ the name ☒ the address ☒ the nationality ☒ the residence

Name and Address LAM, Peter, Kwok, Hing Kløvervang 35 2970 Hørsholm Denmark	State of Nationality GB	State of Residence DK
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

☒ the receiving Office ☒ the designated Offices concerned
☐ the International Searching Authority ☐ the elected Offices concerned
☐ the International Preliminary Examining Authority ☐ other:

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer I. Britel Telephone No.: (41-22) 338.83.38
--	--

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
United States Patent and Trademark
Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 07 August 2000 (07.08.00)	
International application No. PCT/GB99/04424	Applicant's or agent's file reference JDM/P. 401106 WO
International filing date (day/month/year) 23 December 1999 (23.12.99)	Priority date (day/month/year) 24 December 1998 (24.12.98)
Applicant FENN, David, Robert et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:
05 July 2000 (05.07.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was

☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

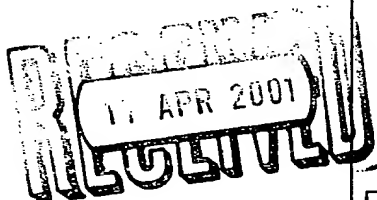
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer S. Mafla Telephone No.: (41-22) 338.83.38
--	---

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
GRANDE BRETAGNE



PCT

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
EXAMINATION REPORT
(PCT Rule 71.1)

Date of mailing
(day/month/year) 12.04.2001

Applicant's or agent's file reference
JDM/P. 401106 WO

IMPORTANT NOTIFICATION

International application No.
PCT/GB99/04424

International filing date (day/month/year)
23/12/1999

Priority date (day/month/year)
24/12/1998

Applicant
PPG INDUSTRIES OHIO, INC. et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Hardy Magliano, N

Tel. +49 89 2399-8151



PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference JDM/P. 401106 WO	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB99/04424	International filing date (day/month/year) 23/12/1999	Priority date (day/month/year) 24/12/1998
International Patent Classification (IPC) or national classification and IPC C08G18/42		
Applicant PPG INDUSTRIES OHIO, INC.et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 05/07/2000	Date of completion of this report 12.04.2001
Name and mailing address of the international preliminary examining authority: <div style="display: flex; align-items: center;"> <div> European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 </div> </div>	Authorized officer Heidenhain, R Telephone No. +49 89 2399 8673



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/04424

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1-11 as originally filed

Claims, No.:

1-12 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
☐ the language of publication of the international application (under Rule 48.3(b)).
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
☐ filed together with the international application in computer readable form.
☐ furnished subsequently to this Authority in written form.
☐ furnished subsequently to this Authority in computer readable form.
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/04424

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-12
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-12
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-12
	No:	Claims	

**2. Citations and explanations
see separate sheet**

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/04424

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Novelty: None of the cited documents discloses polyurethane coating compositions which are the reaction product of diphenyl methane diisocyanate (MDI) and a polyester polyol carrying secondary hydroxyl groups (which are the reaction products of carboxylic acids and epoxides). In DE-A-2 439 276 (D1) reaction products of polyisocyanates and polyols are described, in which the polyol is obtained by reaction of conventional polyester polyols and a minor amount of monoepoxide (up to 0,1 mol monoepoxide per mole OH-groups of polyester). That is, the vast majority of reactive OH-groups are primary OH-groups.

Inventive step: D1 (see above) is considered to represent an adequate closest prior art. The polyurethane compositions in D1 differ from those in the present application in that in the polyols used only a minor proportion of OH-groups are of secondary nature.

Object of the present application is to provide a MDI based, two component coating composition of reduced reactivity. The table on page 10 of the application indicates, that primer coating compositions based on polyester polyols having secondary OH show longer pot life and better hardness development than when using conventional polyester polyols with mainly primary OH (primer 2 vs. primer 3). Although the purpose in D1 was also to achieve a reduction in reactivity, it does not follow from this document that a significant amount of secondary OH apparently is key for achieving this result. Consequently an inventive step can be acknowledged.

**COMPLETE RESPONSE TO WRITTEN OPINION
DATED 6TH SEPTEMBER, 2000
DUE DATE: 6TH DECEMBER, 2000**

30 November, 2000

Despatched: 1 December, 2000

Our Ref: JDM/LB/P. 401106 WO

Your Ref: == == ==

THE EUROPEAN PATENT OFFICE,
International Preliminary Examining Authority,
D-80298 Munich,
GERMANY

VIA FACSIMILE TRANSMISSION
CONFIRMATION: REGISTERED MAIL

Dear Sirs,

Re: International Patent Application No. PCT/GB99/04424
based on United Kingdom Patent Application No. 9828445.8
PPG INDUSTRIES OHIO, INC.
Our File No. P. 401106 WO

I have for reply a Written Opinion dated 6th September, 2000 in connection with the above matter. It is noted that the Examiner takes the view that Claims 1 to 4, 10 and 11 are lacking in novelty in view of DE-A-2439276 (D1) and that Claims 5 to 9 and 12 are lacking in inventive step having regard to D1. The Examiner is respectfully requested to reconsider this opinion in the light of the following points :-

A. NOVELTY

(1) It is to be noted in Claim 1 of the present Application that in the polyesters having secondary hydroxyl groups, the secondary hydroxyl groups are the products of reaction between a carboxylic acid group and an epoxide group. The Examiner appears to believe that in D1 the polyester polyols contain secondary hydroxyl groups which are derived from reaction between a carboxylic acid group and an epoxide group. This is not the case. The intention in D1 is clearly for a small fraction of the hydroxyl groups of a polyester polyol to be converted to secondary hydroxyl groups by reaction with a monoepoxide. In this connection, direction is directed to the first complete paragraph on Page 3 of D1.

(2) It is to be noted that there is no mention in D1 of carboxylic acid groups at all apart from in the example. In this connection, please see below.

Cont/d..... (2)

THE EUROPEAN PATENT OFFICE

(3) The example takes a polyester polyol and reacts this with different quantities of styrene oxide and demonstrates that use of the styrene oxide leads to reduced reactivity with polyisocyanates. It can be seen from the example that the polyester polyol has a hydroxyl value of 54 mg KOH/g and an acid value of 0.56 mg KOH/g. The polyester therefore contains virtually 100 times as many hydroxyl groups as carboxyl groups and in view of this vast excess of hydroxyl groups, it is believed that any reaction between the polyester polyol and the styrene oxide will take place at the hydroxyl groups of the polyester polyol.

(4) The last sentence of the examples supports the position adopted above in specifying that certain percentage additions of styrene oxide mean certain percentages of the available hydroxyl groups have been reacted.

(5) It is therefore submitted that the subject matter of Claim 1 and thus of Claims 2 to 4, 10 and 11 which are dependent on Claim 1, is novel over and above the disclosure of D1.

B. INVENTIVE STEP

(1) D1 does not in any way point the reader towards preparation of polyester polyols containing secondary hydroxyl groups derived from the reaction between a carboxylic acid group and an epoxide group. Indeed in D1, reaction takes place at the hydroxyl groups rather than at the carboxylic acid groups.

(2) The subject matter of D1 is the preparation of polyurethanes. There is however, no mention of coating compositions in D1. In this connection, it is to be noted that the present Application relates to polyurethane coating compositions, their preparation and use.

(3) The compositions disclosed in D1 would be totally unsuitable as a two-component polyurethane composition of the type described in the present Application. For example, D1 mentions adjusting the reaction time in the range of up to 300 seconds (see the second complete paragraph on Page 3). Two-component compositions however, are required to have a pot-life of between 30 minutes and 5 hours as can be seen from Page 2, lines 4 to 6 of the specification of the present Application.

(4) It is therefore submitted that the claims of the present Application are inventive over and above the disclosure of D1.

30 November, 2000
Despatched: 1 December, 2000

- 3 -

THE EUROPEAN PATENT OFFICE

As mentioned above, it is respectfully requested that the Examiner reconsider the conclusions reached in the Written Opinion, and issue a favourable International Preliminary Examination Report.

Yours faithfully,

HUNTINGFORD, DAVID I.
(Professional Representative)
Association No. 105

G:\CLIENT\400-404\P80\401106\WO\P.WPD